



PATENT

Docket No.: 15275/8611 (Dobbins 2-1)

THE UNITED STATES PATENT AND TRADEMARK OFFICE

Reissue Application No.: 08/833,620
Filed: April 7, 1997
U.S. Patent No.: 5,043,002
Granted: August 27, 1991
Patentees: Michael S. Dobbins
Robert E. McLay
For: METHOD OF MAKING FUSED SILICA BY
DECOMPOSING SILOXANES

Examiner:
J. HoffmanArt Unit:
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DECLARATION OF DALE R. POWERS UNDER 37 CFR § 1.132

I, DALE R. POWERS, pursuant to 37 CFR § 1.132, declare:

1. I received a B.S. Degree in Chemistry from Iowa State University in 1970 and a Ph.D. degree in Physical Chemistry from California Institute of Technology in 1975.

2. I hold the position of R&D Fellow at Corning Incorporated. It is my understanding that the above reissue patent application and patent are assigned to Corning Incorporated.

3. I have extensive experience producing a silica preform by oxidation of silicon-containing materials in a flame. In particular, I have worked for 26 years fabricating fused silica glass and optical fibers at Corning Incorporated. My relevant experience includes extensive studies on the rate of reaction of silicon tetrachloride in a reaction flame to make silica soot and the kinetics of silicon tetrachloride oxidation. In addition, I have worked on the use of siloxanes to manufacture optical fiber and high purity fused silica. This work has included the purification of siloxane feedstock materials, the vaporization and delivery of siloxane feedstocks, and the flame hydrolysis of siloxane feedstocks to produce silica soot.

4. I have supervised a series of tests to evaluate the deposition of silica soot produced from vaporized octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane,

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and hexamethyldisiloxane in order to demonstrate the superiority of the former two compounds over the latter one.

5. In carrying out these tests, the siloxane being tested was fed with a metering pump at 2 psi pump pressure into the vertical stack bead bed vaporizer. The vaporizer was maintained at 190°C and a 4.2 SLPM (Standard Liters Per Minute) flow rate of oxygen gas was fed into the vaporizer to provide a vaporized siloxane-oxygen gas mixture which was discharged from the vaporizer through a heated fume line at 180°C and into the center of the silica soot deposition burner. In addition to the vaporized siloxane-oxygen gas mixture flow fed to the silica soot deposition burner, a 10 SLPM flow rate of nitrogen gas was fed to the burner and discharged concentrically around the siloxane-oxygen gas mixture flow leaving the center of the burner face. A 4 SLPM flow rate of oxygen gas was also fed to the burner and discharged from the burner face concentrically around the discharged flow of nitrogen gas. A methane and oxygen fuel mixture gas flow rate of 4 SLPM of methane and 3 SLPM of oxygen was also fed to the burner with the methane oxygen gas mixture leaving the burner face concentrically around the discharged flow of oxygen gas. The burner was positioned 15 cm from the 3/8 inch (9.5 mm) horizontal alumina bait rod target in a soot deposition lathe, with the burner traversing back and forth a 70 cm length of the bait rod as the bait rod was rotated. The gas leaving the burner face was ignited to deposit silica soot on the bait rod. These flow rates were maintained for the 370 minute run time, at which time the deposited soot was measured.

6. 10 cc/minute of octamethylcyclotetrasiloxane (i.e. 3.62 grams of Si atoms per minute) and decamethylcyclopentasiloxane (i.e. 3.63 grams of Si atoms per minute) were delivered to the vaporizer. This flow rate was optimal in its utilization of the soot deposition equipment to achieve stable silica soot production with deposits of high purity and quality. The same flow rate was also used for hexamethyldisiloxane which equates to a level of 2.64 grams of Si atoms per minute delivered to the vaporizer. In each of these cases, a stable flame was maintained, white silica soot was generated, and a white silica soot preform of uniform shape suitable for high purity fused silica glass was produced. The results of these tests were as follows:

Octamethylcyclotetrasiloxane

1205 grams of silica soot deposited
3.26 grams per minute average rate of silica soot deposited

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Decamethylcyclopentasiloxane

1186 grams of silica soot deposited

3.21 grams per minute average rate of silica soot deposited

Hexamethyldisiloxane

855 grams of silica soot deposited

2.31 grams per minute average rate of silica soot deposited

7. In order to achieve an atomic Si delivery rate (i.e. 3.62 grams of Si atoms per minute) of hexamethyldisiloxane which was similar to that achieved above with octamethylcyclotetrasiloxane (3.62 grams of Si atoms per minute) and decamethylcyclopentasiloxane (3.63 grams of Si atoms per minute), a flow rate of 13.71 cc per minute was also used for hexamethyldisiloxane. As a result, the burner flame became unstable, black carbon-laden soot was generated, and a non-uniformly shaped preform was produced. Since this product was unsuitable for high purity silica glass production, the run was terminated.

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8. The results in above paragraphs 6 and 7 demonstrate that silica deposition rates with different siloxane starting materials are most fairly compared at similar volumetric flow rates. As discussed in paragraph 7, when the volumetric flow rate of hexamethyldisiloxane was increased above the 10 cc/minute rate to achieve an Si atom delivery rate consistent with that of octamethylcyclotetrasiloxane and decamethylcyclopentasiloxane, a useful product was not formed. If volumetric flow rates of all siloxanes were reduced to achieve a consistent Si delivery rate, a silica preform would not be formed at efficient production rates. Therefore, the results in paragraph 6 most fairly compare the relative silica deposition of hexamethyldisiloxane with that of octamethylcyclotetrasiloxane and decamethylcyclopentasiloxane.

9. I am familiar with the formation of pyrogenic silicic acid by oxidation of siloxanes in a flame as taught by European Patent Application Serial No. 038,900 to Schwarz et al. ("Schwarz"). Since the use of hexamethyldisiloxane is presented in Schwarz as preferred over other disclosed alternatives, such as polymethylcyclosiloxanes, one would expect hexamethyldisiloxane to achieve better results to the extent, if at all, Schwarz were relevant to making a consolidated glass body. However, as shown in paragraph 6, octamethylcyclotetrasiloxane (and decamethylcyclopentasiloxane) achieved substantially higher silica deposition than hexamethyldisiloxane. In view of Schwarz's disclosure of

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hexamethyldisiloxane as preferred, the results obtained in the above experiments are highly unexpected.

10. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date:

6/30/00Dale R Powers

Dale R. Powers